APPARATUS FOR LARGE-SCALE DIAMOND POLISHING

CROSS-REFERENCE TO RELATED APPLICATIONS

This divisional application claims the benefit of priority to U.S. utility application number 09/541,178, filed in the United States on April 3, 2000, entitled "Method and Apparatus for Large-Scale Diamond Polishing."

FIELD OF THE INVENTION

This invention relates to an apparatus for diamond polishing. More specifically, it relates to the use of plasma-enhanced chemical etching techniques for polishing a synthetic diamond to an optical quality surface.

BACKGROUND

Extreme hardness, high thermal and chemical stability, and optical transparency are properties that render diamonds desirable in numerous optical, electrical, and military applications. In order to overcome the rarity and cost of natural diamonds, synthetic methods of diamond preparation have been developed. Synthetic diamonds are efficiently and cost-effectively fabricated in the form of coatings using plasma-assisted (or plasma-enhanced) chemical vapor deposition (PACVD or PECVD) processes. As deposited, the diamond films are polycrystalline, typically possessing a roughness on the order of 10 to 20 micrometers. The rough surface negates the utility of synthetic diamonds in many applications, particularly optics. When the as-prepared synthetic diamonds are used as lens coatings, for example, their rough surfaces produce excessive

scatter and thus, provide low transmittance. Costly labor-intensive polishing must be performed in order to achieve the required finish for this type of application. Mechanical polishing techniques utilizing diamond paste are typically performed, but because the synthetic diamonds have the same hardness as the diamond in the paste, polishing must be performed repetitively and for an extended period. As a result, the cost of polishing the synthetic diamond to optical quality exceeds the cost of depositing the diamond layer.

To reduce the polishing time and cost, a repetitive ion-implantation mechanical polishing technique was designed and is disclosed in U.S. Pat. No. 5,154,023. When utilizing this technique, the rough diamond surface is first "softened" by the formation of an ion-implanted layer. This softened layer is subsequently subjected to mechanical polishing. The softening and polishing steps are repeated until a desired surface smoothness has been achieved. Each cycle of softening and polishing in this technique affects only a shallow surface layer (on the order of 0.1 microns), so dozens of cycles are necessary to process a typical synthetic diamond to optical quality. The repetitive ion implantation polishing technique requires high ion energies (on the order of 100 keV) in order to achieve ion implantation in the diamond surface; this requirement contributes both to overall cost of the method and also raises potential safety issues. Because the synthetic diamond surface has various grain orientations, line-of-sight effects from the high-energy implantation can result in directional sputtering on the surface, thus hampering the production of a smooth surface. In addition, the ion implantation apparatus typically has a small beam spot and therefore, repeated scanning of the beam over the sample is

necessary to achieve uniform ion-implantation across the surface of a large synthetic diamond.

Synthetic diamond films and wafers have been used in various microelectronic applications, such as heat sinks or substrates for semiconductor devices. In these applications, it is often desirable to impart a specific architecture on the surface of the diamond. Oxygen plasma, coupled with pattern masking, has been utilized to selectively etch synthetic diamond wafers. Typically, the masked wafer is etched in a low-pressure oxygen gas reactor, using electromagnetic radiation to generate an oxygen plasma. Under these conditions, etching of the diamond wafer is rapid compared to conventional mechanical polishing techniques. However, under these conditions, the etching of the wafer is anisotropic, most likely due to physical bombardment by high-energy molecular oxygen ions, so polishing to optical quality smoothness is not feasible.

SUMMARY

One object of the present invention is to overcome the disadvantages of the processes described above by providing an apparatus for rapid, uniform, and cost-effective synthetic diamond polishing. More specifically, one embodiment of the present invention provides an apparatus for effectively polishing a synthetically produced diamond by plasma-enhanced chemical etching using an atomic oxygen polishing plasma source, said source generating high concentrations of low energy atomic oxygen ions over a large surface area. The present invention takes advantage of the ability of low energy atomic oxygen ions to chemically etch a diamond surface at moderate

temperatures. Because the atomic oxygen ions have low energy and high density, they conform to the surface of the synthetic diamond sample, and thus polish the sample with increased uniformity versus known oxygen etching techniques. The rate of polishing is proportional to the density of atomic oxygen, and, in the present invention, this density can be easily controlled by adjusting parameters such as gas pressure, discharge voltage, and plasma ion source power to minimize the processing time.

Because the present invention utilizes a chemical effect to polish the diamond surface, the energy of the atomic oxygen ions generated is much lower than the energy of ions generated during ion implantation techniques or similar high energy beam approaches. Accordingly, the atomic oxygen polishing plasma source disclosed herein can operate at lower voltages than the apparatus for ion-implantation, thereby reducing both capital investment and safety concerns. In addition, because the atomic oxygen polishing plasma source generates a large plume of plasma, large diamond samples can be polished in their entirety without beam scanning, and multiple samples can be polished simultaneously.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic representation of the atomic oxygen polishing plasma source of the present invention;

FIG. 2 is a schematic representation of the plasma composition in the different regions of the atomic oxygen polishing plasma source and vacuum chamber of the present invention;

FIG. 3 is a plot of the atomic oxygen fraction as a function of vacuum chamber pressure at two different discharge voltages in the atomic oxygen polishing plasma source of the present invention;

FIG. 4a is a plot of the fraction of atomic oxygen produced by the polishing source as a function of discharge voltage in an embodiment of the present invention without magnetic filtration;

FIG. 4b is a plot of the fraction of atomic oxygen produced by the polishing source as a function of discharge voltage in an embodiment of the present invention having magnetic filtration;

FIG. 4c is an overlay of **FIG. 4a** and **FIG. 4b** illustrating the contrast between atomic oxygen concentrations in embodiments of the present invention with and without magnetic filtration; and

FIG. 5 is a schematic representation of the chemical etching of a diamond surface by atomic oxygen in accordance with the present invention.

DETAILED DESCRIPTION

The present invention is useful for providing an apparatus for large scale diamond polishing. The following description is presented to enable one of ordinary skill in the art to make and use the invention, which may be incorporated in the context of a variety of applications. Various modifications to the preferred embodiment, as well as a variety of uses in different applications will be readily apparent to those skilled in the art. Notably, the general principles defined herein may be applied to other embodiments; thus, the present invention is not intended to be limited to the embodiments shown, but is to be

accorded the widest scope consistent with the principles and novel features disclosed herein.

A schematic of the preferred embodiment of the atomic oxygen polishing plasma source of the present invention is outlined in **FIG. 1**. The atomic oxygen polishing plasma source **100** is a hollow body, which may be cylindrical, having one open end, henceforth referred to as the plasma source exit **102**, a wall **104**, a closed end **106,** and an interior region **107a** which forms a reaction chamber **107**. The wall **104** and the closed end **106** of the plasma source **100** include a cylindrical array of confinement magnets **108** that are held by their own magnetic fields to the inside surface **110a** of a metal cylinder **110** made of a magnetic material such as low carbon steel. A cooling jacket **111** made of a non-magnetic material such as stainless steel completely encases the cylindrical array of confinement magnets **108.** The apparatus may optionally also include a shield located between the cooling jacket **111** and the reaction chamber **107** made of an oxidant-resistant material such as molybdenum. An electron source **112**, powered by an AC voltage source **114**, is inserted through the closed end **106** into the reaction chamber **107** of the plasma source **100**. For the purposes of clarity, the electron source **112** will be referred to as a filament. However, it will be obvious to one skilled in the art that other electron sources would be suitable in other embodiments. In addition, the filament electron source **112** and plasma source wall **104** are connected to a DC discharge power supply **118** located outside the plasma source. The plasma source is also equipped with a leak valve or gas port **120** at the closed end **106**. A planar array of filtration magnets **122** is located within the plasma source reaction chamber **107,** aligned parallel to the plasma

source exit **102.** A planar transparent electrode grid **126** covers the plasma source exit **102**. The diamond sample, or samples, of interest **130** are placed beyond the plasma source exit **102**.

In the preferred embodiment, the atomic oxygen polishing plasma source is positioned inside an evacuated vacuum chamber **200** (shown in **FIG. 2**). Oxygen gas is introduced into the plasma source **100** through the gas port **120**. The final pressure is selected to maximize the atomic oxygen fraction. The pressure in the reaction chamber **107** is the equilibrium pressure created from the flow between the oxygen leak valve, any gaseous reaction products and the vacuum chamber pump. In one embodiment of the present invention a final vacuum chamber pressure of about 10^{-5} to 10^{-3} Torr provided good results. The electron source filament **112**, which in the preferred embodiment is made of tungsten, tantalum, or iridium, is heated to thermionic temperatures, at which point electrons ranging in energy from approximately 10 to 100 eV are emitted from the filament **112**. The emitted electrons are the primary electrons. When a DC bias voltage from the DC power supply **118** is applied between the filament **112** and the plasma source wall **104**, the primary electrons emitted by the filament are accelerated to the plasma source wall **104**. On their way through the plasma source reaction chamber **107,** the primary electrons collide with the introduced oxygen molecules, producing primarily molecular oxygen ion plasma and secondary electrons ranging in energy from approximately 0 to 3 eV. During the plasma creation process, the cooling jacket **111** cools the plasma source **100** in order to prevent heating of the confinement magnets **108** and filtration magnets **122** and other potentially destabilizing effects.

The resultant plasma is comprised of molecular and atomic oxygen species, both neutral and ionic, and free electrons. An illustration of the plasma composition is presented in **FIG. 2**. Magnetic filtration is used to increase the fraction of atomic ions present in the plasma as follows: The magnetic filter **122** creates a transverse magnetic field in the reaction chamber **107** that prevents the high-energy electrons involved in plasma formation from exiting the plasma source **100**. However, low energy electrons and positive ions can penetrate the filter **122** through a collision diffusion mechanism. In the region **202** downstream of the filter **122**, the low energy electrons aid in the dissociation of the molecular oxygen ions into atomic ions before the plasma leaves the plasma source **100** as a plume **204** on its way to the diamond surface(s) **130**. Dissociation of molecular oxygen ions to atomic ions in the downstream region **202** is dependent on the gas pressure in the vacuum chamber **200**.

In the preferred embodiment, the plume of plasma **204** generated by the atomic oxygen polishing plasma source **100** is of largely uniform density over approximately 12 cm in diameter at the plasma source exit **102,** and broadens with increasing distance from the plasma source exit **102**. Thus, with appropriate positioning from the plasma source exit **102**, the diamond surface(s) **130** may be completely covered by the atomic oxygen plasma plume **204**. Furthermore, because the atomic oxygen ions simply diffuse from the plasma source **100**, the energies of these ions are largely dependent on collision effects, which are controlled primarily by gas diffusion and pressure gradients. As a

result, the atomic ions in the plasma plume **204** have energies less than about 100 eV, rendering them well-suited to chemically etch a diamond sample **130**.

The effect of gas pressure on the fraction of atomic oxygen is shown in **FIG. 3**. The effect of magnetic filtration on the fraction of atomic oxygen present in the plasma in the preferred embodiment is illustrated graphically in **FIG. 4**. As shown in **FIG. 4a**, the fraction of atomic oxygen in this embodiment is on the order of 0.28. The data points in **FIG. 4a** were taken at a pressure of 2×10^{-4} TORR and at a discharge current of 10 Amps. However, as shown in **FIG. 4b**, the addition of the magnetic filter **122** increases the fraction of atomic oxygen to at least 0.7 and in some cases to over 0.94. In **FIG. 4b** the diamond points were taken at a pressure of 4×10^{-4} TORR and a discharge current of 40 Amps, while the square points were taken at a pressure of 4×10^{-4} TORR and a discharge current of 10 Amps. An overlay of the atomic oxygen fraction data with and without the magnetic filter **122** is provided in **FIG. 4c.** In **FIG. 4c**, the diamond points represent the non-filter points shown in **Fig. 4a**, while the square points were taken at a pressure of 4×10^{-4} TORR and a discharge current of 40 Amps, while the triangle points were taken at a pressure of 4×10^{-4} TORR and a discharge current of 10 Amps.

The present invention takes advantage of the ability of atomic oxygen to oxidatively chemically etch diamond surfaces. A schematic of the etching mechanism at a diamond surface **500** is provided in **FIG. 5**. By generating high-density atomic oxygen plasma **502** with magnetic filtration, the atomic oxygen polishing plasma source **100** (shown in FIG. 1) provides for a rapid reaction rate between the atomic oxygen plasma **502** and the

diamond surface **500**. The density, and accordingly the reaction rate, in the preferred embodiment of the present invention can be easily controlled by adjusting the gas pressure, the applied discharge power to the plasma source **100**, and the power to the electron source **112**.

Potential anisotropic effects in diamond polishing are further limited in the present invention by the selective generation of low energy atomic oxygen plasma **502**. Restricting the plasma flow to low energy species limits the physical bombardment of the diamond surface **500** by atomic or molecular oxygen ions, a process that results in directional, and accordingly non-uniform, etching of the diamond surface. Under low energy conditions, the primary etching effect on the diamond surface **500** is the non-directional chemical reaction of atomic oxygen plasma **502** with the diamond surface **500**. In addition, because the atomic oxygen polishing plasma source generates low energy plasma **502**, the safety hazards associated with high energy beams such as utilized in the ion-implantation technique described above are minimized.

Finally, as previously stated, because the diamond is treated in the present invention by a broad plume of plasma **204** (shown in FIG. 2), it is possible to treat a single large sample in its entirety, or several samples **130** simultaneously. The low-energy, high-density atomic oxygen plasma plume **204** diffusing from the source, while already non-directional in its effect, is applied to the entire sample uniformly to create a smooth, optical quality surface. Thus, the line-of-sight effects characteristic of beam polishing are effectively eliminated.